

## **SPECIFICATIONS**

### **Title of Invention :**

Chemical formulations and methods utilizing NPB(n-propyl bromide) as non-aqueous carrier mediums to apply fluorocarbons and other organic chemicals to substrates.

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### **References Cited:**

**U.S. PATENT DOCUMENTS: None**

### **Other References:**

Chem. Abst., 66, 76542m (1967). Fluorine-containing compositions for treating substrates to render them oil-, water- and soil-repellant, comprising A fluorine-containing acrylic copolymer and a fluorine-free poly(meth)acrylate.

## **BACKGROUND OF THE INVENTION**

NPB (n-propyl bromide) has been used in the metal parts degreasing industry, particularly in vapor degreasers for years. Now according to the invention it has been discovered to have a very useful and desired purpose in other industries. Many substrates, for example, home textiles, carpets, upholstery acquire oil-,water- and soil-repellant properties by treatment with fluorocarbons.

These chemicals are now applied to substrates with water based (aqueous) carriers requiring other auxiliary chemicals i.e.: emulsifiers and dispersing agents to keep organics in suspension. These auxiliary chemicals needed for aqueous application often lesson the intended benefit of the applied chemical to the substrate. These aqueous carriers require high temperatures and expensive drying systems to evaporate the water.

Chlorinated hydrocarbons have been used in the past as carrier mediums to apply organic chemicals to substrates when an aqueous carrier could not be used. Chlorinated hydrocarbons are being phased out by mandate of the Environmental Protection Agency (EPA).

## **BRIEF SUMMARY OF THE INVENTION**

By this invention, NPB has shown an excellent alternative to current aqueous and chlorinated hydrocarbons as a carrier medium for application of organics to substrates. NPB is non-regulated, non-toxic and has no ozone pollution properties. NPB is economical and environmentally friendly.

After extensive study, it has been found that the use of NPB as a carrier dramatically improves the performances and durability of benefits achieved by application of organics to substrate and that this invention is superior to current methods and chemistry.

## **DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS**

The invention relates to methods and formulations to provide substrates with treatment to include oil and water repellant treatment, for example; the treatment of home textiles and apparel, which achieve desired effects with significantly smaller amounts of expensive fluorocarbon compounds as Compared to available current technology, as illustrated in Example 2 Compared to Example 4.

The following description, taken in conjunction with the referenced examples, is presented to enable one of ordinary skill in the art to make and use the invention. Various modifications will be readily apparent to those Skilled in the art, and the general principles defined herein may be applied to a wide range of aspects. Thus, the present invention is not intended to be limited to the aspects presented, but is to be accorded the widest scope consistent with the principles and novel features disclosed herein.

Furthermore, the compositions according to the invention should furthermore impart to the substrates, in particular the home textiles, water-repellant actions that meet increased requirements.

Another object comprises providing treatment compositions with which the heat treatment can be carried out at the lowest temperature or, preferably, no heat treatment is necessary (Example 3).

In one aspect, invention relates to substrates from the group consisting of naturally occurring and synthetic textiles and their mixtures, leather, mineral substances, thermoplastic and thermosetting polymers and paper, which are treated with fluorine-containing compositions of the type mentioned

below in an amount of 10 to 10,000 ppm, preferably 50 to 5,000 ppm, particularly preferably 100 to 2,000 ppm, calculated as fluorine and based on the total weight of substrates provided with an oil-, water- and soil-repellant treatment.

In another aspect, other textile auxiliary chemicals can be added during preparation of the treatment formula as according to the invention, or subsequently. Such additives are crease-proofing and soft handle agents, melamine, flame retardant, oleophobicizing agents, hydrophobizing agents, Urethane, finishing agents, extenders for textile auxiliaries and others.

Substrates which are suitable for imparting oil-, water- and soil-repellant properties according to the invention are: linen, cotton, wool, silk, jute, polyamide, polyester, polyacrylonitrile and mixtures thereof, leather, stone slabs, floor tiles, glazed tiles, roof tiles, glass, ground surfaces of silicon, foils and films and compact workpieces of polyolefins, polyesters, polyamides, polycarbonates, polyurethane, polyacetals, polyethers, polysulphides, polysulphones, polyamides and other thermoplastics, as well as of phenol/formaldehyde resins, urea/formaldehyde resins, melamine/formaldehyde resins and other thermosetting resins, paper and

paper-like materials, such as paperboard. Preferred base substrates are home textiles based on naturally occurring and synthetic textiles and their mixtures, which are employed, for example, as carpets, curtains, decorative materials or coverings for upholstered furniture.

Processes for the treatment of such base substrates and therefore for application of the fluorine-containing compositions according to the invention are known to the expert and are, for example, foaming, dipping or spraying of the base substrates; the compositions according to the invention furthermore can be employed during the production of the base substrates, for example the pulp.

Textiles as base substrates, preferably home textiles and apparel can be treated, for example, in the padding, spraying or foaming process.

The padder consists of a liquor trough (chassis) and at least one pair of rubber rolls (Example 2). The textiles to be treated are impregnated with the treatment liquor in the chassis and squeezed off between the rolls; the liquor runs back into the chassis. It is very important that a uniform liquor pick-up is achieved over the entire width of the goods during squeezing-off.

In the padding process, the liquor pick-up is stated in percentage of the weight of goods, and for normal textile constructions can be between 30 and 300%, depending on the quality of the goods and the padder pressure used.

In the spraying process, (Example 3) the textile is sprayed with the treatment liquor. The treatment liquor is finely divided by nozzles and applied uniformly. An amount of treatment liquor precisely defined beforehand is applied to one square meter of textile goods.

In the foaming process, the treatment liquor is continuously foamed mechanically in a commercially available mixer with out the addition of a foaming agent. The foam is produced in the mixing head by mixing the liquor with air. The foam, which emerges, is conveyed via a foam line to a discharge slot in the applicator. The goods are pressed against the slot and taken off via a separate unit, for example a stenter frame. In example 1, a concentration of 98% NPB and 2% Perfluoroalkyl polyacrylate were used. The experiments were carried out on the Gaston Systems, Inc. Foam Applicator, Stanly, NC.

By the invention, it has been discovered surprisingly that a mixture of NPB and Perfluoroalkyl polyacrylate can be foamed with or without the aid of a foaming agent (Example 1). Not using foaming agents greatly improves the benefit of the applied fluorine composition to the substrate and reduces the amount of compound added to fabric to achieve water and oil repellency.

In another aspect, this invention involves the surprising discovery that the use of NPB applied in 100% concentration via dipping and squeezing with pressure rollers (Padding) and the NPB being evaporated away imparts a much improved softness and luster to textile substrates, especially home furnishing, apparel fabrics and upholstery fabrics.

After the treatment, the textiles, preferably home textiles, are dried, it being possible to use temperatures of 120.degree. to 170.degree. C. to achieve the desired treatment effect according to the known procedure. However, good oil-, water- and soil-repellant treatments can also be obtained with the new compositions according to the invention at significantly lower drying temperatures, for example at 25.degree. C (Example 3).

Samples of the materials thus pretreated were taken for testing of the

following effects:

Oil-repellency (according to AATCC 118-1972): The test sample is placed on a horizontal, smooth surface, a small drop (drop diameter about 5 mm) of the test liquids is applied to the test sample with the aid of a dropping pipette, In addition, the sample is evaluated as specified.

The AATCC oil-repellency level of a test fabric is the highest number of that test liquid which does not wet or penetrate into the test material within a time span of 30 seconds. The test liquids and mixtures for the test method are: No. 1: Nujol or paraffin oil DAB 8; No. 2: 65% by volume of Nujol and 35% by volume of n-hexadecane; No. 3: n-hexadecane; No. 4: n-tetradecane; No. 5: n-dodecane; No. 6: n-decane; No. 7: n-octane; No. 8: n-heptane.

Repellency towards a water/alcohol mixture (hydrophobicity): Drops of water/isopropanol mixtures (ratio 90/10 to 10/90) are applied to the test sample. The test result corresponds to the mixture with the highest isopropanol content which remains on the test sample in unchanged form for at least 20 seconds (the value 80/20, for example, is better than 20/80).

## **EXAMPLES**

Compositions which are not according to the invention (Example 4) and which represent the prior art are the following: Nuva HPU (Clariant Corporation). Scotchgard.RTM. FC 396 (3M Comp.) according to DE-A 2 149 292 Baygard.RTM. SF-A . (Bayer AG) according to DE-A 3 307 420 and Zonyl (E.I. Dupont)

The compositions according to the invention (Examples 1, 2 & 3) are non-aqueous solutions contents of which comprise a mixture of NPB (component A) and one or more poly (meth) acrylates (component B).

### **Use of the compositions according to the invention:**

#### **Example 1**

A solution of 98% NPB and 2% Perfluoroalkyl polyacrylate were mixed and applied to the foam generator which imparts the solution to a high speed mixer that generates the solution into foam. The foam was then dispensed to the substrates listed in below via an applicator at 30%-wet pickup. The substrates were then dried at 170 deg C for 1-minute dwell time with the

following results:

Initial					After 10 Home Laundries			
Example 1	Oil	IPA	Spray	Fluoride	Oil	IPA	Spray	Fluoride
Cotton	5	85	100	2160 ppm	2	60	70	1600 ppm
Polyester	6	90	100	1170 ppm	5	90	90	980 ppm
Pes/Rayon	6	80	100		5	60	70	

## Example 2

A solution of 99.6% NPB and .4% Perfluoroalkyl polyacrylate were mixed and applied to the substrates listed below via a pad applicator at 3.5 bars pressure. The solution was applied at noted wet pickup. Again, the substrates were dried at 170 deg C with a 1-minute dwell.

Initial					After 10 Home Laundries			
Example 2	Oil	IPA	Spray	Fluoride	Oil	IPA	Spray	Fluoride
Cotton	6	100	100	2480 ppm	3	90	80	2200 ppm
Polyester	8	90	100	1270 ppm	6	90	90	1100 ppm
Pes/Rayon	8	80	100		6	80	80	

### Example 3

A solution of 99.6% NPB and 2% Perfluoroalkyl polyacrylate were mixed and applied to the substrates listed below via a Spray at 1.5 bars pressure.

The solution was applied at noted wet pickup. Again, the substrates were dried at 170 deg C with a 1-minute dwell.

Initial					After 10 Home Laundries			
Example 3	Oil	IPA	Spray	Fluoride	Oil	IPA	Spray	Fluoride
Cotton	6	85	100	2260 ppm	2	60	70	1690 ppm
Polyester	6	90	100	1170 ppm	5	90	90	1080 ppm
Pes/Rayon	6	80	100		5	60	70	

### Use of the compositions not according to the invention:

#### Example 4

An aqueous Perfluoroalkyl polyacrylate dispersion using Nuva HPU at 2% concentration was prepared and applied via a padding applicator at 3.5 bars pressure. The solution was applied at noted wet pickup. Again, the substrates

were dried at 170 deg C with a 1-minute dwell.

<b>Initial</b>	<b>After 10 Home Laundries</b>
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<b>Example 4</b>	<b>Oil</b>	<b>IPA</b>	<b>Spray</b>	<b>Fluoride</b>	<b>Oil</b>	<b>IPA</b>	<b>Spray</b>	<b>Fluoride</b>
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<b>Cotton</b>	<b>7</b>	<b>100</b>	<b>100</b>	<b>2460 ppm</b>	<b>7</b>	<b>90</b>	<b>80</b>	<b>2210 ppm</b>
<b>Polyester</b>	<b>6</b>	<b>60</b>	<b>100</b>	<b>1270 ppm</b>	<b>4-5</b>	<b>45-50</b>	<b>90</b>	<b>1100 ppm</b>
<b>Pes/Rayon</b>	<b>5</b>	<b>60</b>	<b>100</b>		<b>2</b>	<b>35-40</b>	<b>70</b>	

In all examples, the substrates used were (1) White Polyester 8oz/sq yd (PES), (2) 100% Cotton interlock and (3) 60/40 PES and Rayon Blend.